



NYE COUNTY NUCLEAR WASTE
REPOSITORY PROJECT OFFICE

TECHNICAL PROCEDURE

TITLE: SEM, TEM, ELECTRON MICROPROBE PROCEDURE		Revision: 0 Date: 11-01-00 Page: 1 of 5
PROCEDURE No.: TP-8.7	SUPERSEDES: DRAFT, 02-26-95	
APPROVAL <i>[Signature]</i> <u>12-11-00</u> Project Manager Date	CONCURRENCE <i>[Signature]</i> _____ Principal Investigator Date <i>[Signature]</i> <u>15 DEC 2000</u> Project Quality Assurance Officer Date	

1.0 PURPOSE

The purpose of this procedure is to provide for the collection of SEM (scanning electron microscope), TEM (transmission electron microscope) and Electron Microprobe data of minerals from volcanic rocks and sediments from Yucca Mountain, or other samples from other locations. The methods, procedures and documentation are described. In addition to these analytical instruments, this procedure also provides for the collection of data from x-ray fluorescence (XRF) neutron activation (NA), instrumental neutron activation (INAA), atomic absorption (AA), inductively coupled plasma (ICP) spectrometry, inductively coupled plasma/mass spectrometry (ICP/MS), EDX and WDX dispersive and nondispersive x-ray analysis.

2.0 SCOPE

All or any of these analytical techniques shall be operated by consultant laboratories specializing in chemical analysis and/or materials sciences. Samples that are sent out for analysis will be in



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part prepared by GMI personnel. SEM, TEM and electron microprobe (Probe) analysis will be accomplished with a GMI staff scientist in addition to the subcontracting laboratory staff. Other techniques will be accomplished by sending out the sample and receiving the results. Sample preparation will be completed for SEM, TEM, and Probe samples following the Thin section preparation analysis form and procedure TP-8.2. This procedure provides for sample tracking, calibration, and sample preparation and data reduction.

2.1 APPLICABILITY

This procedure applies to NWRPO principal investigators (PI) performing SEM (scanning electron microscope), TEM (transmission electron microscope, Electron Microprobe minerals and similar analyses for the NWRPO.

2.2 TRAINING

GMI personnel trained in this procedure by the PI shall use this procedure. Outside subcontracting laboratories will use those personnel that they have trained to run their equipment. Training consists of reading this procedure and discussing it with the PI. The preparer of this procedure is deemed as qualified user and can train other personnel to this procedure. Internal standards shall act as quality control items to determine acceptance or rejection of data

3.0 DEFINITIONS

N/A

4.0 RESPONSIBILITIES

- 4.1 The Principal Investigator is responsible for the preparation of this procedure.
- 4.2 The Principle Investigator is responsible for the oversight of the performance of this procedure. GMI personnel are responsible for tracking the samples by use of a Transfer of Custody Form for a Split Sample, Thin Section Request Form, TP-8.2, and SEM, TEM and Electron Microprobe Analysis Form.
- 4.3 The subcontracting laboratory is responsible for alignment and calibration of the equipment being used for analysis. That (those) laboratory(s) is (are) responsible for tracking the samples and records in its (their) possession.

5.0 PROCESS

Consultant laboratory operational procedure for the equipment used shall be noted in the laboratory notebook, and on the SEM, et al analysis form. Duplicate analyses of samples will be run to ascertain reproducibility of results. Laboratories will supply GMI with support data on their QA programs. Standards used by the laboratory shall be described. These data need to be recorded by the PI and submitted to the NWRPO QA Records Center.

Equipment - The equipment used shall be described as to make and model and to settings used to acquire the information. This information shall be noted in the laboratory notebook and the analysis form.

Sample Preparation -Samples are prepared for analysis by different means depending upon each analysis performed. For each sample analyzed the detail method for sample preparation will be recorded in the SEM, TEM and Electron Microprobe Analysis Form and in the SEM, et al Laboratory Notebook. Procedures in place that deal with sample preparation should be so noted. Sample numbers shall be registered with each analysis.

Handling, Shipping and Storage Requirements -A unique identifier number shall be attached to each and every sample. When samples are shipped a list of the sample numbers for the samples in the shipment shall be checked. The receiving laboratory will fill out a sample transfer of custody form, a copy of which will be returned by FAX upon receipt of the samples. Samples returned with laboratory data shall also be recorded. After laboratory analysis is complete all sample material will remain in storage. SEM, et al Analysis Form shall also track the location of each sample.

Calibration - All equipment is calibrated in accordance with normal consultant laboratory practices. These shall be documented in the laboratory notebook.

5.1 Standards

5.1.1 EDS

Secondary Standards

Spectra will be acquired for 300 seconds in at least 3 spots using 20-micron raster on each mineral grain with dead-time limited to between 10 and 40 %. Spectrum data will be provided.

EDS Acceptance/ rejection criteria - The sample will be deemed unacceptable for use as a standard if any elements are determined to be present that are not reported in the analysis or indicated by stoichiometry.

Primary Standards

Primary standards used will be so noted in the laboratory notebook

5.1.2 WDS

Primary standards will be used. Secondary standards will be unacceptable.

5.1.3 Other Analyses

Primary standards will be used where applicable. They shall be recorded in the laboratory notebook and on analysis forms.

5.2 Data Reduction

All laboratory information received concerning the morphology, textural data and geochemical data obtained for the samples studied will be analyzed by the PI and Co-PI's. This information will be provided in data reports, among other documents. Data reduction methods will be reported in the laboratory notebook and in the reports.

Special research needs may require deviations from this procedure. The operator will document any and all deviations in the laboratory notebook and on the analysis form.

5.3 Accept/reject Criteria - Limitations

Quantitative elemental analysis requires precise sets of conditions that are difficult to acquire. All analysis is to be considered **semiquantitative** and is to be presented as such in any and all reports.

Data is accepted when it tests proper to be reproducible, and has conforming standards that have been accepted.

6.0 REFERENCES

NWRPO Quality Assurance Program Plan

7.0 RECORDS

Consultant Laboratory QA Program Documentation
Data on Standards
Equipment Operation Descriptions
Analysis Forms
Laboratory Notebooks
Transfer Of Custody Forms
Thin Section Request Form
Photomicrographs
Tiff Files
Graphs and Tables from Instrument Printouts
Certified Analysis Sheets
FAX -Transfer Of Custody Form for samples receipt

8.0 ATTACHMENTS

A: SEM, TEM, and Electron Microprobe Analysis Form

SEM, TEM, and Electron Microprobe Analysis Form

Sample number: _____ Organization: _____

Name: _____ Date: _____ Date Sent to Lab.: _____

TYPE OF ANALYSIS: _____

NAME OF LABORATORY: _____

Date data returned: _____ Date samples returned: _____ Samples used up _____

Sample Custody Form: _____

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Description of equipment and settings:

.....
Description of sample preparation:

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Standards used:

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Calibrations information:

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Type(s) of records and data recovered:

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Data reduction information:

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Limitations and Remarks: